Characterization Study of Chitosan - Carbon Nanotube Bead as Metal Ions Adsorption

By

MUHAMAD MUJAHID BIN IBRAHIM

Dissertation submitted in partial fulfillment of the requirement for the Bachelor of Engineering (Hons)

Chemical Engineering

JULY 2005

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CERTIFICATION OF APPROVAL

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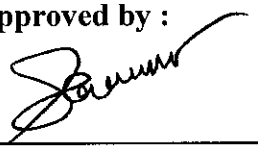

(Puan Asna Mohd Zain)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsibility for the work which had been done during this project except as being specified in the references and acknowledgments. All the original work contained in this project have not been undertaken or done by unspecified sources of persons

(MUHAMAD MUJAHID BIN IBRAHIM)

CHEMICAL ENGINEERING

ABSTRACT

In this final year research project, the research and experiment will be conducted in order to study the characteristic of chitosan-CNT in adsorbing metal ions. The comparison will be made between raw chitosan and chitosan gel in their efficiency in absorbing metal ions. The objective of the research can be summarized as below:

- To extract chitosan from raw material which is crab shell and determined weight percentage of the biopolymers that can be extracted.
- To construct new material which is based on chitosan and carbon nanotube this later will be used as one of the absorber.
- To study the efficiency of these absorber in adsorbing metal ions from industrial waste water

Generally, heavy metal ions are very dangerous for our health and also to the environment. Those heavy metal ions such as Zinc, Plumbum, nickel, cadmium and others can give a long term effect for our health. Nowadays, these metal ions are extensively found in most of the waste water from the industrial area. Regarding to the environmental issues, this project was done to determine the efficiency of the absorber in absorbing heavy metal ion from the industrial waste water.

The methodology of this project starts with the extraction of the chitosan from raw material which involved several processes such as demineralization, deproteination and deacetylation. Chitosan which had been obtained will be further processed in order to produce chitosan gel and chitosan-carbon nanotube gel. The equipment will be used in determining metal ions in the solution will be FAAS.

From the process which had been done, 70.81 gram of chitosan have been extracted from crab shells powder and from the data analysis, it shows that the carbon nanotube had improves the efficiency in metal ions adsorption by 16 percent compared to chitosan and 91 percent compared to chitosan gel.

ACKNOWLEDGEMENT

Alhamdullilah, finally I had completed my final year research project with duration of 15 weeks. I could not have gone this far without the help and support from so many people around me.

My biggest gratitude forwarded to Puan Asna Mohd Zain, my supervisor for her guidance and help throughout the project which had been done. I am so thankful for her guidance and encouragement which had been given during this final year project.my gratitude also goes to AP Dr Norani Muthi for her guidance and sample of CNT which had been given toward the final year project.

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CHAPTER 1
INTRODUCTION

1.1 Background

The principal objective of metal ion removal process is generally to remove any dangerous metal ions that can harm human being, affect the environment and nature. Before the technology of using chitosan as a metal ion removal is implemented, metal ions are removed by using resins especially in water treatment process.

Chitosan also is widely used for many applications since it has a unique characteristic and it is categorized as a biopolymer material (Refer to Table 1.1). As a glucose amine biopolymer, chitosan selectively absorb transition metal ions but it doest absorbed alkali and alkaline earth metal ions. This is the main reason why chitosan is very ideal material used for the metal ions removal agent.

Table 1.1 Application of chitosan

Industry	Applications
Food industry	Removal of dye, suspended solid. Color stabilization, animal feed additive.
Medical	Bandages, blood cholesterol controller, contact lens, etc.
Biotechnology	Enzyme immobilization, protein separation, cell recovery, chromatography, cell immobilization
Agriculture	Seed coating, fertilizer, controlled agrochemical release.
Cosmetics	Moisturizer, face, hand and body creams, bath lotion.
Membrane	Permeability control reverses osmosis membrane.

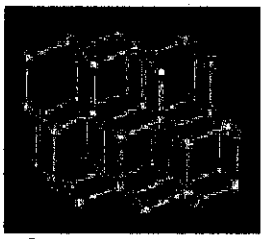
In this project, crab shell was used as a main source of chitin because it contains more chitosan compare to other mollusk found in the market. Chitosan is able to form a complex with metal ions in waste water because of the high content of amine and carboxyl group.

In order to find a higher percentage adsorption toward metal ions, chitosan must be combined with other material. In this scope of project, the material selected was carbon nanotube (CNT).

The main reason why CNT are choose:

- **It has a smaller size**
- **It has high degradation temperature or melting point.**
- **High tensile strength**
- **Stable at high temperature**

CNT can be divided into 2, which is multiwall and single wall carbon nanotube(See appendix B).CNT which is a new carbon allotrope is slightly different with the other structure of carbon material such as diamond, graphite and c60 bulky ball (As seen in figure 1.1) . For this project, a multiwall CNT will be used as it is easier to be produced compared to single wall CNT.

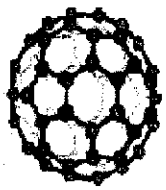


Diamond sp^3 Bonding, hard and insulating

(a)

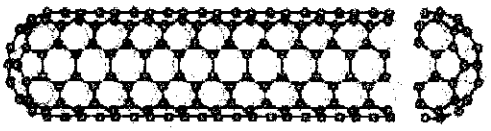
Graphite: sp^2 Bonding
Soft between graphene layers

(b)



C_{60} "bucky-ball": hollow sphere
~$1x10^{-9}$ m (1nm) in diameter

(c)



Carbon Nanotube: 1-50nm in diameter, 10 - 100 micrometer long

(d)

Figure 1.1 Carbon allotrope (a) diamond bonding, (b) graphite (c) hollow sphere (d) Carbon Nanotube (CNT)

1.2 Problem Statement

Most of the hazardous heavy metal ion present in the waste water is coming from the chemical and petrochemical industries. Since these heavy metal ions are very harmful to our health and can give a big impact to the environment, the issues have become a major concern. Since chitosan is a very fragile material, soft and easily be degraded in a high temperature, a combination of chitosan-CNT is believed to be feasible as a bead for a metal ions adsorbent as it will slightly change the physical properties of the chitosan

As a result, the standard discharge limit of these metals under Environmental Quality act 1974 of Malaysia(sewage and industrial effluents)1979 are kept equivalent below 1.0 mg/l in which the standard for iron is 1.0 mg/l , 0.1 mg/l for lead and 1.0 mg/l each for zinc, nickel, copper and chromium accordingly.

Concern to the environmental issues we are facing nowadays, this final year project has chose to study on the metal ion removal in industry waste water by generating an effective adsorbent consist of CNT and chitosan.

1.3 Objectives and Scope of Study

Objectives of the project are;

1) To extract chitosan from crab shell and determine weight percentage of the biopolymers that can be extracted.
2) To study the characteristic of chitosan-CNT and its capability in adsorbing metal ions.
3) To construct a new material is which a combination of chitosan-CNT.

From chitosan-CNT which had been produced, several experiments will be done in order to determine:

- The efficiency of the chitosan-CNT gel in adsorbing heavy metal ions compared to chitosan and chitosan gel only.
- Effect of temperature in the preparation of chitosan - CNT gel.

This project required some experiment in order to achieve the objectives. In order to determine the effect of pretreatment toward the metal ions uptake, various solutions of waste water had been used. In this project, several adsorption methods will be used in order to make sure the chitosan can be successfully combined together with the CNT.

Further study of this experiment is to analyze the efficiency of chitosan–CNT gel after being treated at different temperature.

CHAPTER 2
LITERATURE REVIEW AND/OR THEORY

2.1 History of Chitosan

In 1859 *chitosan* was discovered by Rouget. While experimenting with chitin, he observed that the compound could be manipulated via chemical and temperature treatments to become soluble. Ledderhose, in 1878 determined chitin to be made of glucosamine and acetic acid. It was not until 1894 that Hoppe-Seyler named the modified chitin, chitosan. Early 20^{th} century research often involved sources of chitin including fungi and crab shells. The work of Rammelberg in the 1930's eventually led to the confirmation of the identity of chitin from these sources. By hydrolyzing chitin in different manners, Purchase & Braum determined that chitin is a polysaccharide of glucosamine and by 1948; Matsusshima was able to obtain a patent for producing glucosamine from crab shells.

The use of chitosan for heavy metal ion retention was first reported by Muzzarelli. Later, Muzzarelli and Rocchetti demonstrated that particles with smaller porous size possess a greater available surface to retain adsorbate. If the sample is porous, the greater area is given by the surface inside the porous and the external surface will be scorned. The decrease in grain size will have no effect in the adsorption area[14].

The usage biopolymers as an adsorbent in removing the heavy metals are applicable nowadays. This kind of adsorbent is much cheaper than any other type of adsorbent. The heavy metals are removed by applying the adsorption process where the experiment is conducted in a fixed bed of carbon nanotube- chitosan.

2.2 Chitosan and Chitin

Chitosan is derived from a natural product, chitin, quite abundant in shrimp's shells. Chitin is a non toxic, biodegradable polymer of high molecular weight. After cellulose, it is the most common polysaccharide found in the nature. Like cellulose, chitin is a fiber, and in addition, it presents exceptional chemical and biological qualities that can be used in many industrial and medical applications.

Chitin and chitosan have the same chemical structure(As seen in Figure 2.1).Chitin is made up of a linear chain of acethylglucosamine groups. Chitosan is obtained by removing enough acetyl groups (CH3-CO) for the molecule to be soluble in most diluted acids. This process, called deacetylation, releases amine groups (NH) and gives the chitosan a cationic characteristic. This is especially interesting in an acid environment where the majority of polysaccharides are usually neutral or negatively charged.

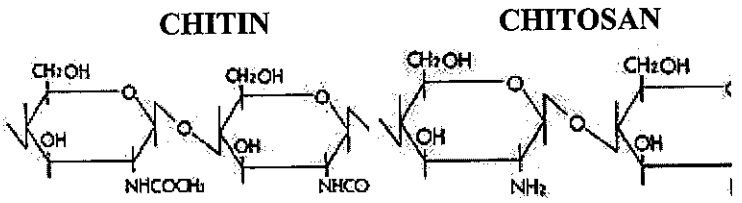


Figure 2.1 Chemical structures of chitin and chitosan

2.3 Current Application

In current application of chitosan in ion removal technology, chitosan is treated with several method and way to produce a porous magnetic bio polymers beads and known as cross linked chitosan beads.

This chitosan beads is prepared by drop wise addition of an aqueous solution containing chitosan and acetic acid into a bath of aqueous NaOH. The NaOH neutralizes the acetic acid and precipitates the chitosan as a gel, which assumes the form of a bead. Linear glucosamine chains within the gel bead are chemically cross linked with glutaraldehyde dissolved in water. Cross linked gel beads are freeze dried to remove water in a way that makes the material highly porous (Figure 2.1).

This cross linked chitosan beads are designed for a high metal ion adsorption capacity and has a chemical stability needed for surface water and subsurface groundwater treatment. This material is cross linked with glutaraldehyde, possesses internal surface areas exceeding 200 m^2/g and insoluble in acid solution (Figure 2.2). The chitosan beads also contain magnetite, therefore porous magnetic chitosan beads can be retrieved from the site clean up by devices such as magnetic field gradient separator [6].

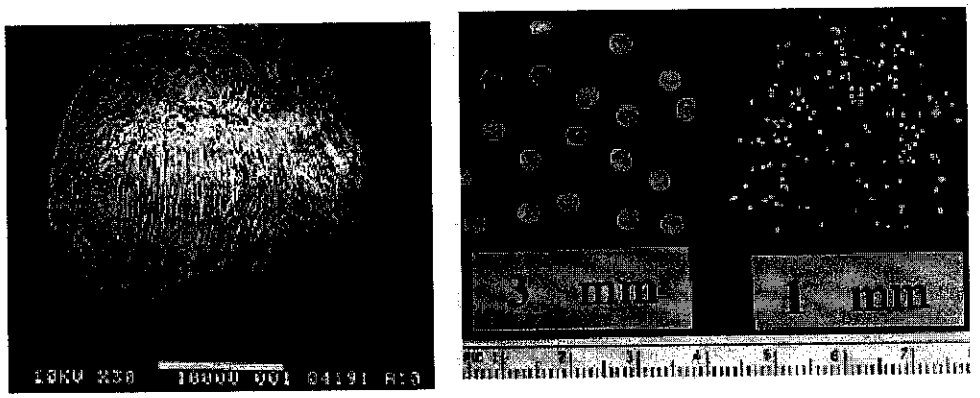


Figure 2.2 Cross linked chitosan

CHAPTER 3
METHOLOGY AND PROJECT WORK

3.1 Brief Procedure

The project is carried out to study the characteristic of chitosan-CNT as a metal ions removal unit. Before that, chitosan and carbon nanotube must be prepared first before experiment can be done.

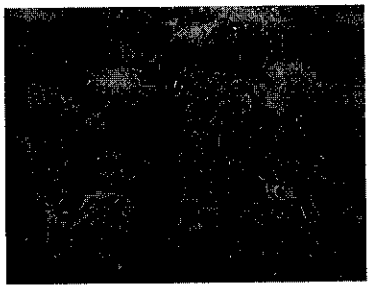
3.1.1 Preparation of crab shells sample

In this project, chitin will be extract from the crab shell before chitosan can be obtained. Two kilogram of crab was bought from the market. The crab then was washed down to remove any dirt or sludge before it will be well processed. All the flesh and the inner past of the crabs were removed and the shells is taken and washed to remove any flesh which was attached to it. The flesh removing process was quite difficult as the crab was quite small and hard. Knife and hammer were used to break the shells apart before the flesh can be taken out. The shells was dried for two days in order to remove unpleasant odor smell and to make it more brittle before grinding process can be done on lab.

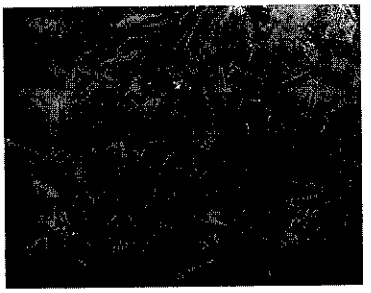
For the next step, the shells will be grind in order to produce a fine powder of crab shell. This was to ensure a large surface area when we want to react it with other chemical in the decarbonation, deproteination and deacetylation process. After the grinding process, the powder will be heat up in an oven up to 120^{o} degrees Celsius for about 2 hours. Heating process will make sure the powder will become more brittle and to prevent organic decomposition.

3.1.2 Procedure in preparation of crab shells powder.

- The crab shells was dried up under sun for 2 days in order to remove all the odor smells and remove all the water on it.
- Shells was crushed into a smaller pieces by using hammer or crucible before it can be grind using a lab grinder or mortar grinder. This was to make sure a large total surface area in chemical reaction between HCL and NaOH.
- The fine powder then was sieve using sieve machine for about 2 minutes in order to obtain a very fine powder.
- After that, the powder was placed into oven at 120° degrees Celsius for 2 hours in order to make sure it become more brittle and to prevent organic decomposition. Figure 3.1 shows the prepared crab shells



a) Inner shell of crab



b) Shell of crab leg

c) Outer shell of crab

Figure 3.1 Crab shells components, (a) Inner shell of crab tissue, (b) Crab leg and (c) Outer shell of crab

3.1.3 Preparation of Chitosan

Generally, quality of chitosan varies with the degree of deacetylation of the N-acetyl groups, molecular weight, purity, manufacturing process, color, clarity, consistency and uniformity. What will be discussed on this section is some of the method used to extract chitosan from shell of mollusk, shells of insects, crustaceans, and fungal biomass [2].

One of the most simplest and conventional way in preparation of chitosan consisted of extracting the substance from crab shells or other mollusk shells. However, this method will only produce a low quality of chitosan in a certain amount.

By using method of Alimunar and Zainuddin, chitin was extracted from a crab shells after certain process being done to the shells powder. In the early step of the process, crab shells were taken by removing all the flesh from a fresh crab.

3.1.4 Extraction of Chitin using Broussignac method (1970).

In order to obtain chitin from a crab shells, 2 main processes must be done which is decarbonation and deproteination process. Decarbonation process was a process done in order to remove all the calcium carbon contained in the crab shells. This process was done by mixing crab shells with hydrochloric acid which will produce a quite amount of calcium salts. During this process, carbon dioxide also will be produced, and this can be seen by a formation of foams.

Deproteination process is a process where covalent chemical bond has to be destroyed between the chitin, protein complexes in order to form free radicals. Free radicals are the active sites of the molecules which in presence will enhance the growing of molecular chains of biopolymers.

The use of NaOH will result in partial deacetylation of chitin and deproteination of the polymers that may lower the molecular weight of the chitin. The powdery sample obtains after deproteination process was composed of chitin together with intermediated amount of chitosan.

3.1.5 Procedures of extraction of chitin.

- 20 ml of 1.5 M of hydrochloric acid was added per gram of shells powder. This process will neutralize the shell powders by removing calcium carbonate constitute in the organic compound in the shells.
- The mixture was kept for 2 days with a constant stirring at room temperature. Most of the solution then was decanted in the end of the mixing process by using filter paper.
- After that, 20ml of NaOH was added per gram of Decarbonated crab shells powder. The mixture was heated for 1 hour using water bath at temperature of 90 degrees Celsius.
- The mixture was left to react for 1 day at room temperature
- After the reaction complete, the powdery residue was collected by using Buchner funnel, washed down using distilled water and dried using acetone.
- Finally, the sample was left overnight to let the weight stabilize and the total weight of chitin extracted was measured and recorded.

3.1.6 Calculation on preparation of 1.5M HCL and 1.2 M NaOH solutions.

The quantity required for preparation of 1.5 M solution of HCL can be calculated using equation below:

$$m_1V_1 = m_2V_2 \quad \text{..... Equation (1)}$$

Where,

At HCL density of 36%, $m_1 = 1.19$ kg/l

Volume of pure HCL, $V_1 = x$

At density of 1.5 M HCL, $m_2 = 0.05469$ kg/l

Volume of 1.5 M HCL, $V_2 = 500$ ml

Calculation of m_2:

m_2 = Morality x molecular weight of HCL

= 1.5 M x 36.46

= 0.05469 kg/l

V_1 can be calculated:

$m_1V_1 = m_2V_2$

(1.19 kg/l)(V_1) = 0.05469 kg/l (0.5 l)

V_1 = 22.97 ml

From the calculation, m_2 0.05469 kg/l and V_1 is 22.97 ml.

Preparation of NaOH 1.2 M solution.

For the preparation of sodium hydroxide (NaOH) 1.2 M solution, we need to calculate how much NaOH pellet need to be used for preparation of 500 ml of NaOH solution.

Concentrations of NaOH want to be prepared: 1.2 mol/l .

Mass of NaOH needed in preparing the solution can be calculated by using equation:

$M = n/v$..... Equation (2)

$n = v * M$.....Equation (3)

Where the volume of solution to need to be prepared is 500ml, concentration of NaOH is 1.2 mol/l.

No of mol NaOH can be calculated using equation below:

$n = m /MW$..... Equation (4)

$m = MW * n$.....Equation (5)

Substitute equation 3 into equation 5

m = M x v x MW

= 1.2 mol/l x 0.5 l x 40 g/mol

= 24 g of NaOH

From the calculation, 24 gram of NaOH pellet is be used in order to prepare 500 ml of 1.2 M NaOH solution.

3.2 Deacetylation of Chitin to Chitosan Using Method of Alimunar and Zainuddin

Referring to the method of Alimunar and Zainuddin, 1992 [12], chitosan will not be formed at alkali concentration of 45%. A higher alkali concentration will lead to a faster rate if deacetylation thus, 50 ml concentrated solution of NaOH was used and being mixed up with each gram of chitin obtained.

The concentrated solution was prepaid by mixing 50 g of NaOH in 100 ml of distilled water. The process was done in a fume board as a safety precaution. After the complete reactions of deacetylation process, the solution was dilute before filtration to avoid the concentrated solution from dehydrating the filter paper and change the composition in the chitosan powder with other component in filter paper. The residue of the mixture was collected and washed with distilled water and dried using acetone wash.

3.2.1 Procedure in Deacetylation of chitin

- 50% NaOH solution was prepared by mixing 50 g of NaOH pallets in 100 ml of distilled water.
- The solution prepared then was heat in the water bath at 95 degrees Celsius for 3 hours. A 50 ml of concentrated solution was added per gram of chitin and left for 3 days at room temperature.
- Most of the solution was removed by pour it away and then all the residue was added with 1 liter of distill water.
- The mixture was added with 2% of acetic acid until it almost neutralize before it can be filtered.
- The powdery residue was collected using Buchner funnel and dried using acetone wash. Figure 3.4 show chitosan obtained from deacetylation process from chitin.

Figure 3.2: Chitosan

3.3 Preparation of Chitosan – CNT gel [5]

A chemically modified electrode based on chitosan-CNT coated glassy carbon electrode has been developed by a researchers with the main function is to determine dopamine (DA) and ascorbic acid (AA) simultaneously [5].This method for the preparation of Chitosan – carbon nanotube material is discovered by Lingyan Jiang etc; July 2004. The main function of this material, which they had been constructed, is as a modified electrode for simultaneous detection of dopamine and ascorbic acid.

3.3.1 Procedure in preparation of chitosan gel:

- 0.55 gram of chitosan was put in a 500ml beaker.
- 30 ml of 1.2M acetic acid was pour into the beaker containing the chitosan. The solution was stir and heat up at 50 degrees Celsius for 15 minutes in order to make sure the chitosan was well mix and diluted in the acid.
- The solution was chilled down up to room temperature.1.2M NaOH solution was added to the chitosan solution. White precipitate will be formed as the acid being neutralized by the alkali.
- The mixture was filtrate in order to obtain the chitosan gel.
- Chitosan gel was put in a Petri dish and being dried until most of the water being removed from the gel.

3.3.2 Procedure in preparation of chitosan-carbon nanotube gel:

- 0.55 gram of chitosan was put in a 500ml beaker.
- 30 ml , 1.2M acetic acid was pour into the beaker containing the chitosan
- 0.01 gram of carbon nanotube was mixed with the mixture
- The solution was stir and heat up at 40 degrees Celsius for 15 minutes in order to make sure the chitosan was well mix and diluted in the acid.
- The solution was chilled down up to room temperature.
- 1.2M NaOH solution was added to the chitosan solution.
- White precipitate will be formed as the acid being neutralized by the alkali.
- The mixture was filtrate in order to obtain the chitosan gel as seen in Figure 3.3 and 3.4 accordingly.
- Chitosan gel was put in a Petri dish and being dried until most of the water being removed from the gel.
- The procedure was repeated at 3 different temperatures which is at 50, 60, and 70 degrees Celsius.

The method of preparation chitosan-CNT discovered by Lingyan Jiang is mainly not for an ion metal absorption purpose; those, some modification should be made to increase the adsorption rate and capacity of the chitosan-CNT.

A part from that, it is proposed that the CNT being mixed up with a solution of the cross linked chitosan solution. One of the reasons is because cross-linked chitosan is originally designed for a metal ion removal unit which had a high porous magnetic structure [6].

Figure 3.3: Chitosan carbon nanotube gel

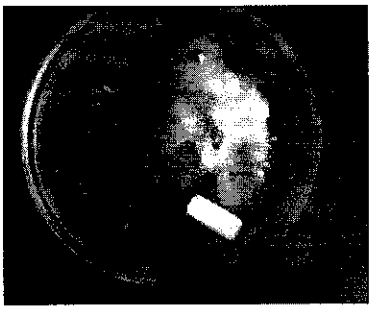


Figure 3.4: Chitosan gel

An experiment should be done in order to observe the advantage of the new material created, which is the combination of CNT and cross linked chitosan solution. During this project, an experiment will be done in order to determine:

- The efficiency of chitosan -CNT in adsorbing heavy metal,
- Effect chitosan-CNT which treated at different temperature in adsorbing metal ions.

A chemical modification on chitosan may be justified by two objectives [8]:

- Preventing the dissolving of the polymer when metal sorption is performed in acidic solution.
- Metal sorption properties might be improved (increase of sorption capacities or enhancement of sorption selectivity)

3.4 Preparation of carbon nanotube: [3]

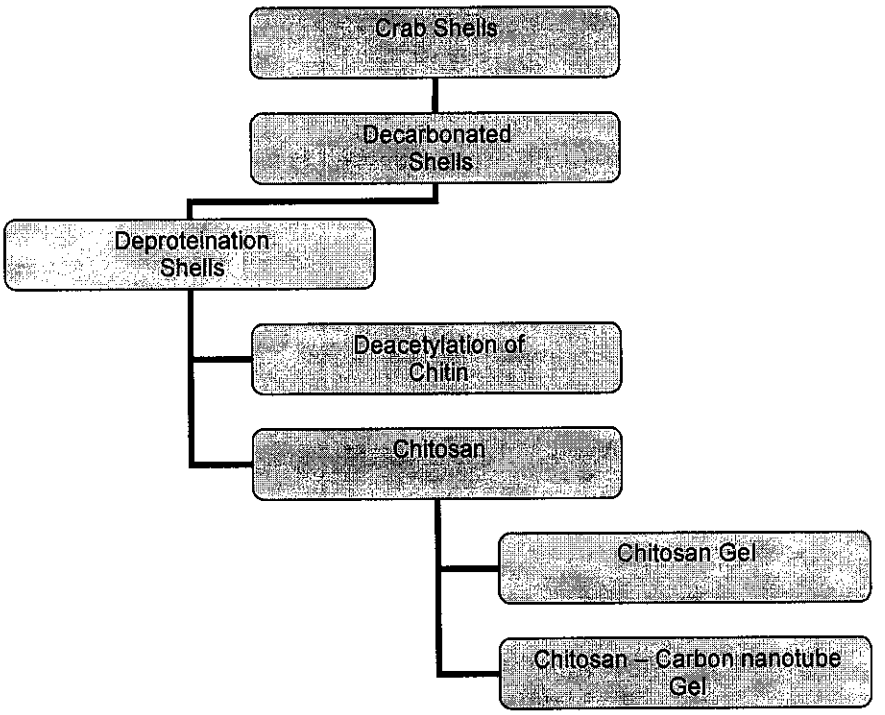
There are several methods employed to make CNT, such as arc discharge, laser ablation, and chemical vapor deposition. By using chemical vapor deposition process (CVP), a large quantity of carbon nanotube can be produced at a lower cost (compared to the other method), high purity, while tuning of physico-chemical properties like surface structure, diameter, and morphology [3]. This process is done by reacting carbon containing gas such as acetylene, ethylene, ethanol, etc. with a metal catalyst particle (usually cobalt, nickel or iron) at temperatures above 600°C.

By using CVD equipment, several conditions can affect the CNT production rate of the carbon deposits .the effect of reaction temperature, catalyst particle size and shape, chemical nature of the catalyst and the reactant composition can give a significant effect in producing carbon nanotube.

3.4.1 Procedure [3]: Preparation of catalyst for CNT:

- The alumina-supported Ni–Fe alloy catalysts with molar ratio of 1:1 were prepared by means of homogeneous deposition–precipitation with iron cyanide, which was used to ensure a uniform composition of the individual catalyst particles.
- By using the iron containing precursor $Na_2Fe\ (CN)_5NO$ and nickel in the form of $Ni(NO_3)_2$, a Ni–Fe ratio of 1:1 can be obtained.
- Normally, the aqueous iron cyanide solution was injected slowly into a vigorously stirred suspension of alumina (γ-Al_2O_3, BET 189.6 m^2/g, pore volume 0.29 cm^3/g) in an aqueous solution of nickel nitrate.
- The pH of the suspension was adjusted to 5 with nitric acid. After addition, the loaded support was filtered, washed, and dried at room temperature in vacuum for 24 h.
- It was then calcined at 600 °C in 100 ml/min flowing air for 10 h and sieved to 75–120 μm granule. Loading of the catalysts was adjusted to 20 wt.%.
- The prepared catalyst was characterized by XRD to analyze the phases and particle size distribution. The XRD study was performed using a Siemens D5000 X-ray diffractometer.
- The resulted peaks were analyzed with a profile fitting program (SIEMENS DIFFRAC^{plus} PROFILE), and a crystal size and micro strain software (DIFFRAC^{plus} WIN-CRYSIZE).
- Before XRD characterization the calcined metal oxides were reduced to the metallic state in a 1:1 N_2/H_2 mixture (total flow rate 200 ml/min) for 15 h at 600 °C.
- After the reduction step, the samples were cooled down to room temperature under flowing N_2 and then passivated in a 4% air/N_2 mixture (totally 100 ml/min) for 2 h to prevent bulk oxidation of the bimetallic particles.

The following diagram summaries for preparation of the adsorbent chitosan, chitosan gel and chitosan CNT.



3.5 Industrial waste water

In order to obtain a suitable waste water solution that contain sufficient amount of metal ions in the experiment, 10 waste water sample was taken and being analyze in order to trace any metal ions on it.

In this experiment, the absorbent efficiency toward Zinc, Plumbum and Ferum absorption will be determined. A part from that, a suitable amount of zinc ions were need in the waste water solution. From the 10 sample taken, the most suitable waste water for the experiment was collected from the final outlet drain of battery factory.

3.6 Flame Atomic Absorption Spectrometry

Flame atomic absorption is a very common technique for detecting metals and metalloids in environmental samples. It is very reliable and simple to use. The technique is based on the fact that ground state metals absorb light at specific wavelengths. Metal ions in a solution are converted to atomic state by means of a flame. Light of the appropriate wavelength is supplied and the amount of light absorbed can be measured against a standard curve.

The technique of flame atomic absorption spectroscopy (FAAS) requires a liquid sample to be aspirated, aerosolized, and mixed with combustible gases, such as acetylene and air or acetylene and nitrous oxide. The mixture is ignited in a flame whose temperature ranges from 2100 to 2800 °C.

During combustion, atoms of the element of interest in the sample are reduced to free, unexcited ground state atoms, which absorb light at characteristic wavelengths, as shown in figure 3.5.

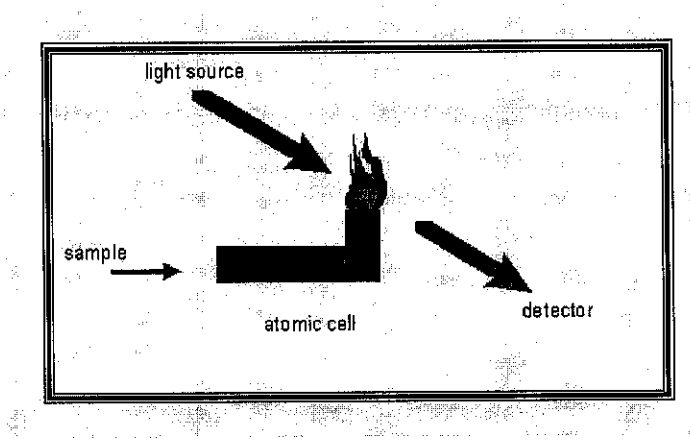


Figure 3.5: Operation principle of an atomic absorption spectrometer

The characteristic wavelengths are element specific and accurate to 0.01-0.1nm. To provide element specific wavelengths, a light beam from a lamp whose cathode is made of the element being determined is passed through the flame. A device such as photon multiplier can detect the amount of reduction of the light intensity due to absorption by the analyte, and this can be directly related to the amount of the element in the sample.

Flame atomic absorption hardware is divided into six fundamental groups that have two major functions: generating atomic signals and signal processing. Signal processing is a

growing additional feature to be integrated or externally fitted to the instrument. The instrument parts are shown in Figure 3.6.

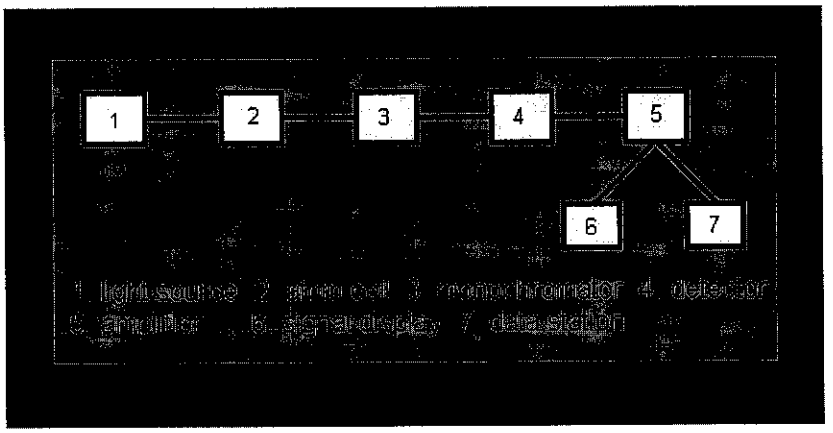


Figure 3.6: Schematic of basic instrumental parts of atomic absorption spectrometer

A cathode lamp (1), is a stable light source, which is necessary to emit the sharp characteristic spectrum of the element to be determined. A different cathode lamp is needed for each element, although there are some lamps that can be used to determine three or four different elements if the cathode contains all of them. Each time a lamp is changed, proper alignment is needed in order to get as much light as possible through the flame, where the analyte is being atomized, and into the monochromator.

The atom cell (2), is the part with two major functions: nebulization of sample solution into a fine aerosol solution, and dissociation of the analyte elements into free gaseous ground state form. Not all the analyte goes through the flame; part of it is disposed as shown in the figure.

As the sample passes through the flame, the beam of light passes through it into the monochromator (3). The monochromator isolates the specific spectrum line emitted by the light source through spectral dispersion, and focuses it upon a photomultiplier detector (4), whose function is to convert the light signal into an electrical signal.

The processing of electrical signal is fulfilled by a signal amplifier (5). The signal could be displayed for readout (6), or further fed into a data station (7) for printout by the requested format.

3.6.1 Procedure of Metal Ion Analysis for Industrial Waste Water Sample

To analyze the metal ions contained in the waste water solution, the waste water sample needs to be tested using the FAAS equipment.

Procedure:

1. The standard solution for metal to be tested, Zinc, Plumbum, and Ferum were prepared by dilution method. The concentrations for the standard solution were 5ppm, 10ppm and 15ppm for Zinc, Ferum and Plumbum.
2. 5ml of the standard solution was put in the test tube that was provided.

Note: Deionized water must be used in preparation of standard solution to obtain a correct calibration curve.

3. 5 ml of each waste water sample were placed in the test tube and put in the test tube holder in the FAAS.
4. The FAAS equipment was ran in the automatic mode to detect the selected metal ions. All the data obtained was printed for further analysis.

The procedure was repeated for other metal ions. From the data obtained, we can calculate the metal ions uptake by the absorber by knowing the final concentration of the solution after it being treated by the absorber.

It is very important to obtain a good and linear calibration graph before any data obtain from the FAAS can be used for the further analysis. To obtain a good calibration curve, the standard solution must be prepared carefully and the dosage used in the dilution must be correct and accurate which can be achieved by using micropipette.

3.7 Study of Metal Ion Removal Efficiency

The next step for this project was to determine the efficiency of this 3 adsorbent in adsorbing metal ions at different time in contact and the efficiency of the chitosan – CNT gel treated at different temperature in absorbing metal ions at different time in contact.

3.7.1 Absorption Rate of Metal ion Removal by Using Chitosan, Chitosan Gel, Chitosan – Carbon Nanotube Gel.

1) 3 Samples of industrial waste water sample were prepared in 150ml beaker.
2) 0.55 gram of chitosan, chitosan gel which contained 0.55 gram of chitosan and chitosan-carbon nanotube gel which contained 0.55 gram of chitosan was added in each of the beaker.
3) The samples were places under continuous stirring at room temperature and pH 7.
4) 15 ml of the sample was taken at 15min, 30 min, 1 hour, 1.5 hour, 2 hours, 3 hours, 4 hours, 6 hours, 8 hours and 10 hours.
5) Final concentration of the metal ions that was leftover in the treated water was determined using FAAS.
6) Graph of absorption capacity versus time was plotted. The efficiency of the absorber can be determined from the time taken for the absorber to reach the maximum capacity in absorbing metal ions.

3.7.2 Absorption Rate of Metal ion Removal by Using Chitosan – Carbon Nanotube Gel treated at different temperature.

1) 4 Samples of industrial waste water sample were prepared in 150ml beaker.
2) Chitosan-carbon nanotube gel which contained 0.55 gram of chitosan and being treated at 40, 50, 60, 70 degrees Celsius was added in each of the beaker.
3) The samples were places under continuous stirring at room temperature and pH 7.
4) 15 ml of the sample was taken at 15min, 30 min, 1 hour, 1.5 hour, 2 hours, 3 hours, 4 hours, 6 hours and 8 hours
5) Final concentration of the metal ions that was leftover in the treated water was determined using FAAS.
6) Graph of absorption capacity versus time was plotted. The efficiency of the absorber can be determined from the time taken for the absorber to reach the maximum capacity in absorbing metal ions.

3.8 Test for chitosan

Purification of chitosan was attempted by dissolution in 1% ethanoic acid. The filtrate was collected leaving impurities behind as residue. Standard bench NaOH was added to precipitate the chitosan as a white gelatinous precipitate. The Van Wisselingh test (Tracy, 1955) is the most accurate test for chitosan When I_2-KI solution was added, the precipitate turned brownish red. The mixture was then acidified with H_2SO_4, upon which the chitosan immediately turned black. By successfully testing for chitosan, we could indirectly infer that chitin was present in the processed powder.

3.9 List of equipment used during the experiment

In order to perform the above experiment, the equipment required is:

- Beaker
- Buchner funnel
- Volumetric flask
- Petri dish
- Filter paper
- Mortar grinder
- Sieve
- Oven
- Flame atomic absorption spectrometer

CHAPTER 4
RESULT

4.1 Extraction of chitosan and metal ions content

From the experiment which had been done, on the early stage, 20 contained 2.625 chitosan had been obtained from 150 gram of crab shell powder(2.6 gram of chitosan per batch).There were still about 60 gram of crab shell powder which had been not processed yet because lack of apparatus and equipment. The experiment was done continuously in order to process as much as possible crab shells powder to obtain chitosan (see appendix A).

Table 4.1 show the chitin and chitosan extraction rate from crab shell and table 4.2 shows the initial concentration of metal ions from battery manufacturer waste water.

Table 4.1: Total weight percentage of chitosan obtained through out the process.

Stages	Crab shell powder	Chitin	Chitosan
Weight (g)	197	99.74	70.81
Wt %	-	50.63	71.00

Table 4.2: The initial concentration of selected metal ions in waste water from battery factory.

Metal Ions	Untreated waste water Concentration (PPM)
Plumbum	37.8361
Ferum	6.4098
Zinc	5.8917
Nickel	0.1232
Chromium	0.1123

4.2 Zinc adsorption

Zinc adsorption capacity by chitosan at different contact time is shown in Table 4.3.

Table 4.3: Result of Zn^{2+} adsorbed by 0.55 gram of chitosan at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
0.25	5.8917	5.7549	0.1368	2.32
0.5	5.8917	5.6543	0.2374	4.03
1	5.8917	4.8483	1.0434	17.71
1.5	5.8917	4.7675	1.1242	19.08
2	5.8917	4.5436	1.3481	22.88
3	5.8917	4.2629	1.6288	27.65
4	5.8917	4.0426	1.8491	31.38
6	5.8917	3.8418	2.0499	34.79
8	5.8917	3.742	2.1497	36.49
10	5.8917	3.7421	2.1496	36.49

Zinc adsorption capacity by chitosan gel at different time in contact is shown in Table 4.4.

Table 4.4: Result of Zn^{2+} ions adsorbed by chitosan gel contained of 0.55 gram of chitosan at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
0.25	5.8917	5.7551	0.1366	2.32
0.5	5.8917	5.6628	0.2289	3.89
1	5.8917	4.9883	0.9034	15.33
1.5	5.8917	4.775	1.1167	18.95
2	5.8917	4.6556	1.2361	20.98
3	5.8917	4.4342	1.4575	24.74
4	5.8917	4.0926	1.7991	30.54
6	5.8917	3.8818	2.0099	34.11
8	5.8917	3.7428	2.1489	36.47
10	5.8917	3.7422	2.2095	37.50

Zinc adsorption capacity by chitosan-CNT gel at different time in contact is shown in Table 4.5. The adsorption chitosan –CNT gel which treated at different temperature is shown in Table 4.6, 4.7, 4.8 and 4.9 for temperature of 40, 50, 60 and 70 accordingly.

Table 4.5: Result of Zn^{2+} ions adsorbed by chitosan-CNT gel contained 0.55 gram of chitosan at different time in contact

Time (Hour)	Initial concentrati (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
0.25	5.8917	5.7543	0.1374	2.32
0.5	5.8917	5.6454	0.2463	4.03
1	5.8917	4.8332	1.0585	17.71
1.5	5.8917	4.6925	1.1992	19.08
2	5.8917	4.3512	1.5405	22.88
3	5.8917	4.0411	1.8506	27.65
4	5.8917	3.7409	1.9508	31.38
6	5.8917	3.7411	2.1506	34.79
8	5.8917	3.7412	2.1505	36.49
10	5.8917	3.7409	2.1508	36.49

Table 4.6: Result of Zn^{2+} ions adsorbed by chitosan-CNT gel contained 0.55 gram of chitosan treated at 40 degrees Celsius at different time in contact

Time (Hour)	Initial concentratio (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
1	5.8917	4.8333	1.0575	17.95
1.5	5.8917	4.6926	1.1982	20.34
2	5.8917	3.9313	1.9595	33.26
3	5.8917	3.7409	2.1499	36.50
4	5.8917	3.7404	2.1504	36.50
6	5.8917	3.7392	2.1516	36.52
8	5.8917	3.7393	2.1515	36.52

Table 4.7: Result of Zn^{2+} ions adsorbed by chitosan-CNT gel contained 0.55 gram of chitosan treated at 50 degrees Celsius at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
1	5.8917	4.8233	1.0675	18.12
1.5	5.8917	4.6916	1.1992	20.36
2	5.8917	3.9213	1.9695	33.43
3	5.8917	3.7419	2.1489	36.48
4	5.8917	3.7504	2.1404	36.33
6	5.8917	3.7352	2.1556	36.59
8	5.8917	3.7373	2.1535	36.56

Table 4.8: Result of Zn^{2+} ions adsorbed by chitosan-CNT gel contained 0.55 gram of chitosan treated at 60 degrees Celsius at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
1	5.8917	4.8233	1.0675	18.12
1.5	5.8917	4.6826	1.2082	20.51
2	5.8917	3.9313	1.9595	33.26
3	5.8917	3.7407	2.1501	36.50
4	5.8917	3.7404	2.1504	36.50
6	5.8917	3.7387	2.1521	36.53
8	5.8917	3.739	2.1518	36.53

Table 4.9: Result of Zn^{2+} ions adsorbed by chitosan-CNT gel contained 0.55 gram of chitosan treated at 70 degrees Celsius at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
1	5.8917	4.8433	1.0475	17.78
1.5	5.8917	4.7016	1.1892	20.19
2	5.8917	3.9213	1.9695	33.43
3	5.8917	3.7411	2.1497	36.49
4	5.8917	3.7504	2.1404	36.33
6	5.8917	3.7372	2.1536	36.56
8	5.8917	3.7373	2.1535	36.56

4.3 Plumbum ions adsorption

Result of Plumbum ions adsorbed using chitosan at different time in contact is shown in table 4.10.on the other hand, adsorption capacity for similar ion by chitosan gel at different time contact is tabulated in table 4.11.a combination of chitosan-CNT gel performance to adsorb Plumbum ions at different time in contact is shown in Table 4.12.

Table 4.10: Result of Pb^{2+} ions adsorbed by 0.55 gram of chitosan at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
1.5	37.8361	29.6282	8.2079	17.16
2	37.8361	29.8117	8.0244	16.77
3	37.8361	29.9619	7.8742	16.46
8	37.8361	19.5075	18.386	38.44
10	37.8361	13.6463	24.1898	50.57

Table 4.11: Result of Pb^{2+} ions adsorbed by 0.55 gram of chitosan gel at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
1.5	37.8361	32.353	5.4831	14.49
2	37.8361	32.7089	5.1272	13.55
3	37.8361	32.6811	5.155	13.62
8	37.8361	29.3613	8.4748	22.40
10	37.8361	24.8348	13.0013	34.36

Table 4.12: Result of Pb^{2+} ions adsorbed by 0.55 gram of chitosan-CNT gel at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
1.5	37.8361	13.2904	24.5457	64.87
2	37.8361	12.7399	25.0962	66.33
3	37.8361	9.1865	28.6496	75.72
8	37.8361	9.0253	28.8108	76.15
10	37.8361	9.0419	28.7942	76.10

4.4 Ferum ions adsorption

The result of Ferum ions adsorption capacity by chitosan (Table 4.13), chitosan gel (Table 4.14) and chitosan-CNT (Table 4.15) at different time in contact.

Table 4.13: Result of Fe^{2+} ions adsorbed by 0.55 gram chitosan at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
0.5	6.4098	6.0535	0.3563	5.56
1	6.4098	5.3003	1.1095	17.31
1.5	6.4098	4.6591	1.7507	27.31
2	6.4098	4.2657	2.1441	33.45
3	6.4098	4.2069	2.2029	34.37
4	6.4098	4.1999	2.2099	34.48
6	6.4098	4.2251	2.1847	34.08

Table 4.14: Result of Fe^{2+} ions adsorbed by chitosan gel which contains of 0.55 gram of chitosan at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
0.5	6.4098	5.9863	0.4235	6.61
1	6.4098	5.4081	1.0017	15.63
1.5	6.4098	4.7291	1.6807	26.22
2	6.4098	4.3007	2.1091	32.90
3	6.4098	4.2447	2.1651	33.78
4	6.4098	4.2167	2.1931	34.21
6	6.4098	4.2293	2.1805	34.02

Table 4.15: Result of Fe^{2+} ions adsorbed by chitosan-CNT gel which contains of 0.55 gram of chitosan at different time in contact

Time (Hour)	Initial concentration (ppm)	Final concentration (ppm)	Absorbent capacity (ppm)	Percent uptake (%)
0.5	6.4098	5.9695	0.4403	6.87
1	6.4098	5.3031	1.1067	17.27
1.5	6.4098	4.7739	1.6359	25.52
2	6.4098	4.3203	2.0895	32.60
3	6.4098	4.1859	2.2239	34.70
4	6.4098	4.1551	2.2547	35.18
6	6.4098	4.1369	2.2729	35.46

CHAPTER 5
DISCUSSION

5.1 Total amount of Chitosan extract from crab shells, sample preparation of chitosan gel and chitosan carbon nanotube gel.

From the preparation of chitin which involved with decarbonation and deproteination process, 99.74 gram of chitin had been obtained from 197 gram of crab shells powder. From the deacetylation process, 70.81 gram of chitosan had been obtained. The yield of chitosan obtained from the deacetylation process was about 71.00 weight percent.

The value is quite high compare to the previous final year student. Based on previous final year project reported, only 67.74 weight percent was yielded from the experiment. The yield of chitosan is very dependent on the process in preparation the samples. Any human error might affect the purity and amount of chitosan which can be obtained. In order to avoid lost of chitosan during filtration process after demineralization, deproteination and deacetylation, Buchner Funnel was used in collecting the residue. Using an ordinary filter paper might cause some of sample losses .Tweezer was used in order to collect some of the sample which is highly bond to the filter paper. This process was done carefully to avoid the filter paper from shredded due to overpressure.

During the decarbonation process, it can be clearly seen that white form of bubble was release due the reaction of hydrochloric acid with the calcium carbonated contained in the crab shells. Calcium carbonate was produced from the reaction and it must be decomposed after the end of the decarbonation process. During the deproteination process, the mixture of crab shells and NaOH had change its color from red brownish to white pale after it being heated and let to react for 1 day. The color was changed because all the protein in the crab shell had been reacted with NaOH.

In the preparation of chitosan gel, chitosan mixture, which can be produced by dissolving acetic acid with chitosan, was let to react with sodium hydroxide in order to

produce a white precipitate which does not dissolve in water. The white precipitate was collected carefully and let to be dried to remove most of the water inside it.

From the observation, the volume of gel which had been obtained during the preparation of chitosan -carbon nanotubes gel was slightly more compared to the chitosan gel even though same amount of chitosan was used in the preparation of these samples. One of the theory is because the carbon nanotube inside the mixture had give more surface area and create a highly porosity structure inside the gel.

5.2 Initial concentration of selected metals ions present in industrial waste water

Table 4.2 shows the initial concentration of metal ions present in the waste water sample collected from acid lead factory located near Batu Gajah industrial area. Hypothesis that can be made was those waste water will be contain more Plumbum compare to other metal ions since those factory was handling a Plumbum material in their operation. from the result obtain from the AAS, it shows that concentration of Plumbum was about 37.8361 ppm followed by 5.8917 ppm of Zinc and 6.4098 ppm_of Ferum. The other metal ions such as nickel, and chromium were detected below 1 ppm.

5.3 Efficiency of metal ion removal by chitosan, chitosan gel and chitosan-CNT gel at different contact time.

In this experiment, chitosan, chitosan gel and chitosan-CNT gel was used in determine the efficiency in adsorbing metal ions. Chitosan, chitosan gel, and chitosan carbon nanotube gel was added in 100 ml of industrial wastewater with a constant stirring.1 beaker was used for every adsorbent and sample were taken at 10 different time intervals , which at 15min, 30min, 1, 1.5, 2.0, 3.0 , 4.0 , 6.0 , 8.0, 10.0 hours. The samples were filtrated first to remove any solid particle before it can be analyze using AAS to prevent clogging in the AAS equipment. For this experiment, the temperature was set to be constant at room temperature with a pH value of 7.

5.3.1 Absorption of heavy metal ions at various time contacts

From Figure 5.1, 5.2, 5.3 it shows the time taken for the absorber to reach the maximum capacity of the zinc ions which had being absorbed. From the graph, we can clearly see that the time taken for the chitosan-CNT gel adsorption rate is higher compared to chitosan and chitosan gel. The maximum capacity also seems to be higher compared to chitosan and chitosan gel.

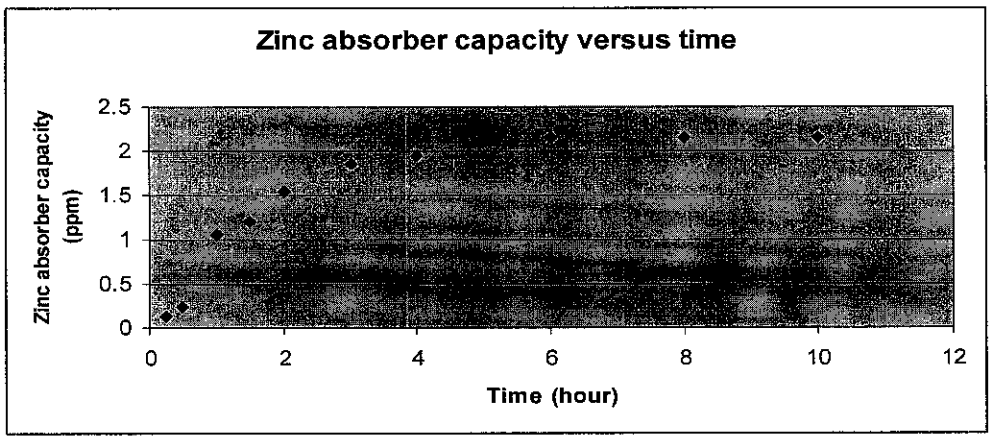


Figure 5.1: Zinc adsorption capacity for chitosan-carbon nanotube gel

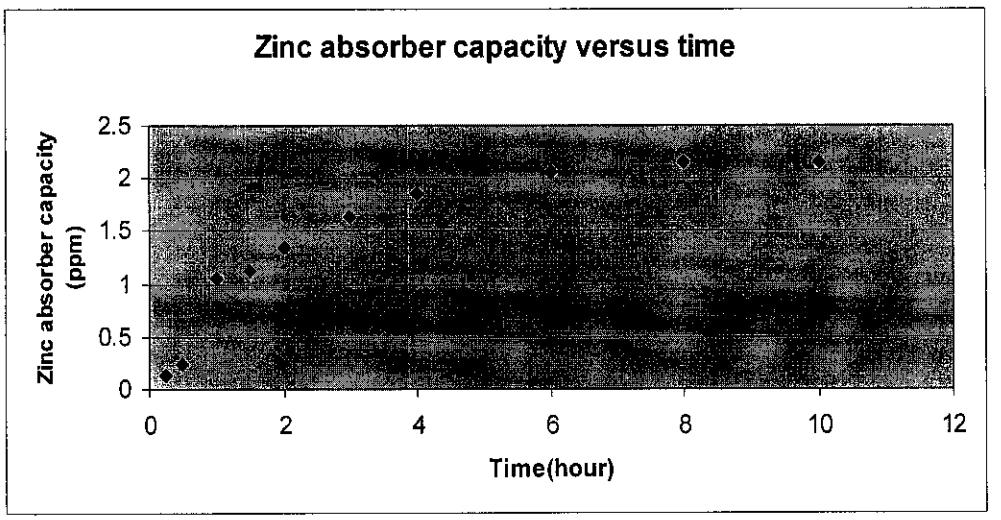


Figure 5.2: Zinc adsorption capacity for chitosan

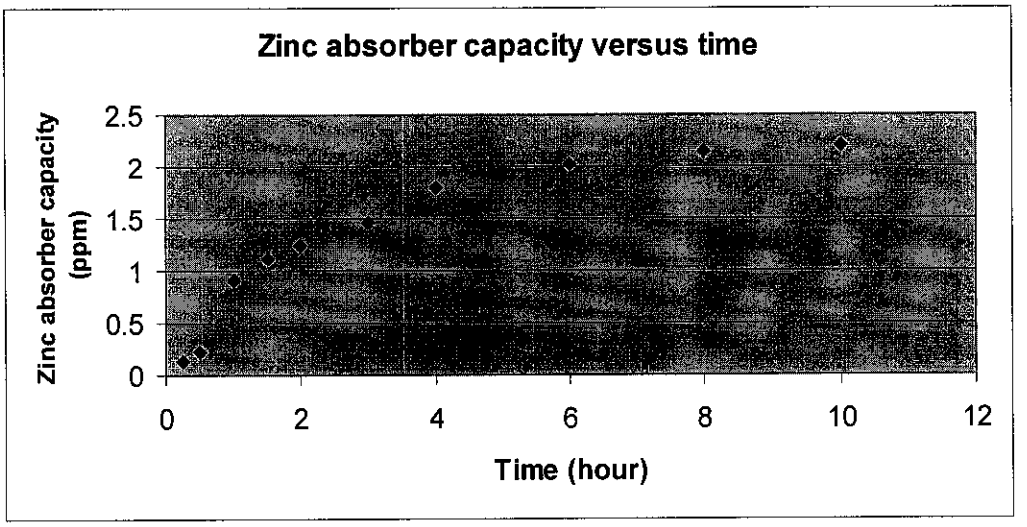


Figure 5.3: Zinc adsorption capacity for chitosan gel

One of the reasons why this might be happen is because, the presence of the carbon nanotube particle inside the gel had increase the surface area of the absorber and also it had increase the porosity of the absorber in absorbing metal ions. High porosity will make the ions easier to reach the centre side of the absorber.

Absorption rate of chitosan gel can be seen slightly lower than chitosan. it is because, during the dehydration process, where water will be remove from the gel, the porosity of the gel will be decreased due to the change in gel structure.

The maximum capacity for chitosan carbon nanotube gel was at 2.1508 ppm of zinc ions, while for the chitosan and chitosan gel was at 2.1496 and 2.2195 ppm. From the result, we can see that maximum uptake of zinc ion for these 3 adsorbent is quite lower because most of the adsorbent had adsorbed other metal ions rather than zinc ions.

From Figure 5.4, 5.5, and 5.6 it show the time taken for the absorber to absorb Plumbum ions. From the graph, we can clearly see that most of the ions had been absorbed especially for the CNT gel .The rate of absorption and the efficiency for the carbon nanotube gel is slightly higher compared to chitosan and chitosan gel.

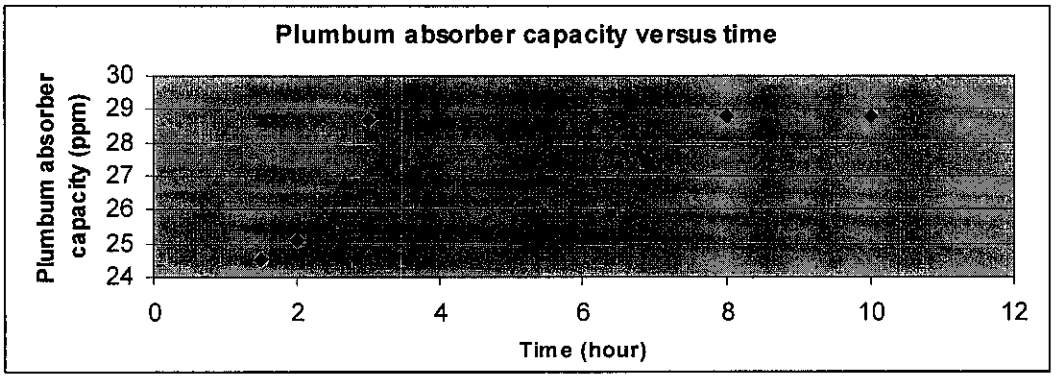


Figure 5.4: Plumbum adsorption capacity for chitosan carbon nanotube gel

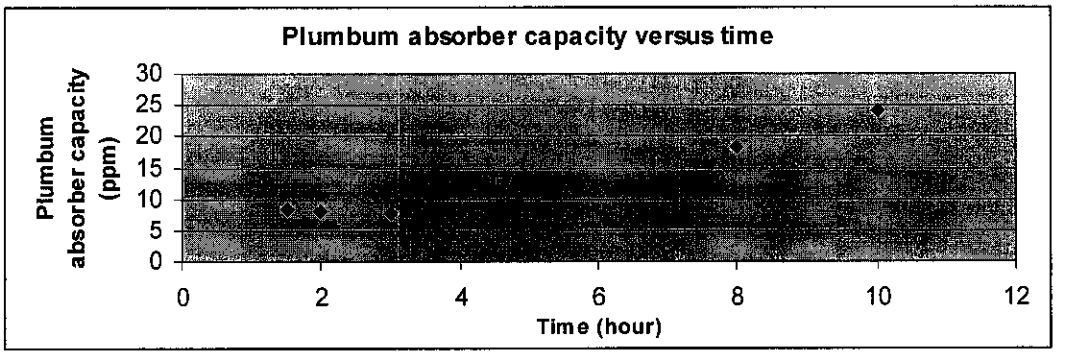


Figure 5.5: Plumbum adsorption capacity for chitosan

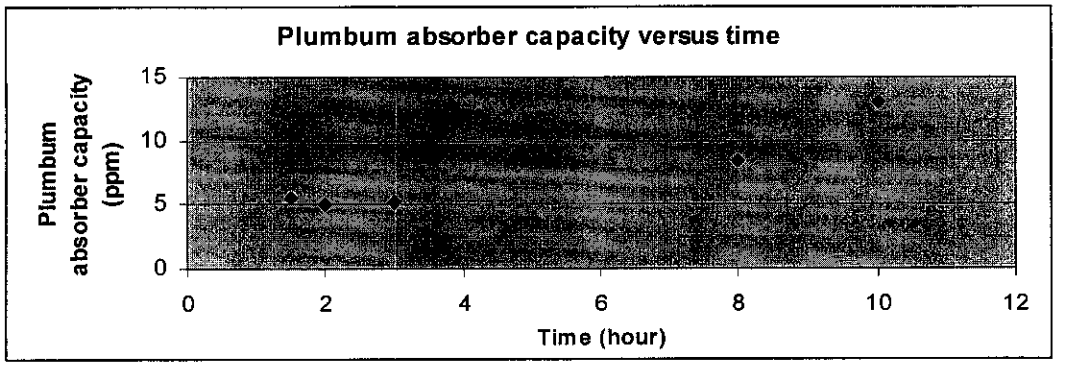


Figure 5.6: Plumbum adsorption capacity for chitosan gel

From Figure 5.7, 5.8 and 5.9, it show the time taken for the adsorbent in adsorbing Ferum ions .From the graph, the trend is quite same . One of the major factor is because the interferences between other concentration ions such as Plumbum. The maximum capacity and rate of adsorption for the adsorbent also quite same because of interference that might be happen during the adsorption process.

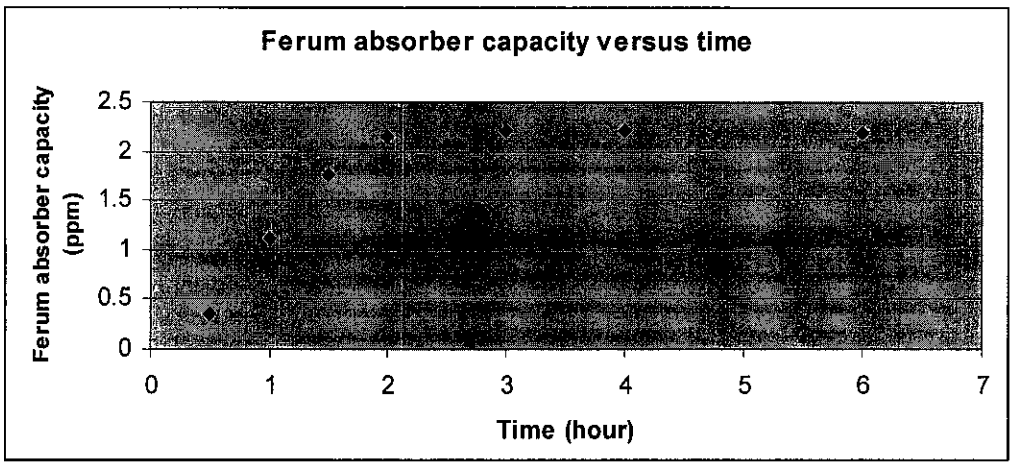


Figure 5.7: Ferum adsorption capacity for chitosan-carbon nanotube gel

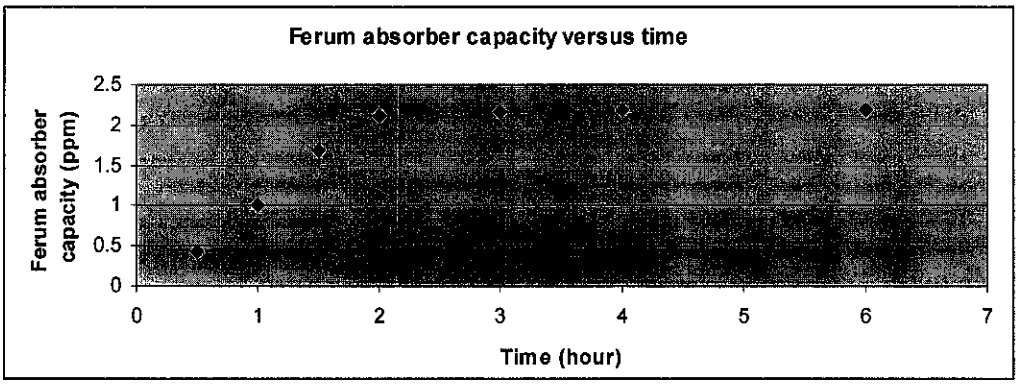


Figure 5.8: Ferum adsorption capacity for chitosan gel

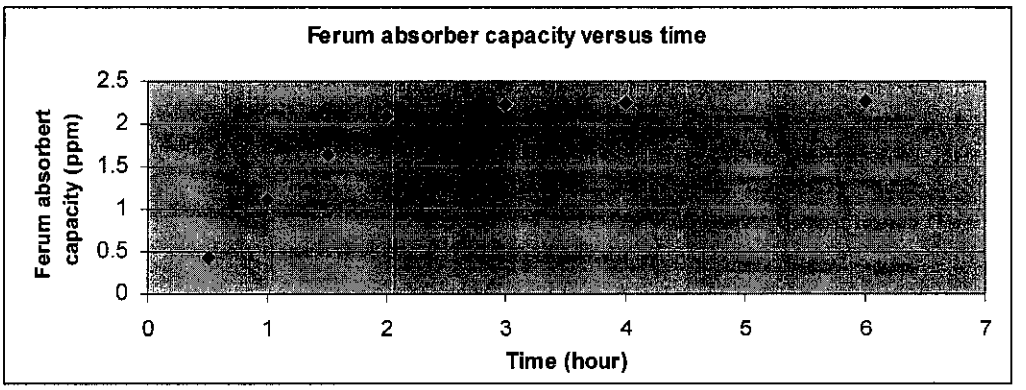


Figure 5.9: Ferum adsorption capacity for chitosan

5.4 Efficiency of metal ion removal by chitosan – carbon nanotube gel treated at different temperature

In this experiment, zinc ions were used to observe the effect of different temperature treatment in the preparation of chitosan carbon nanotube gel. Four samples were produced, which had been treated at 40, 50, 60 and 70 degrees Celsius. From Figure 5.10, 5.11, 5.12 and 5.13, it shows the maximum capacity for the adsorbent in adsorbing zinc metal ions. There was no significant effect found on the adsorption capacity of chitosan-CNT prepared at different temperature.

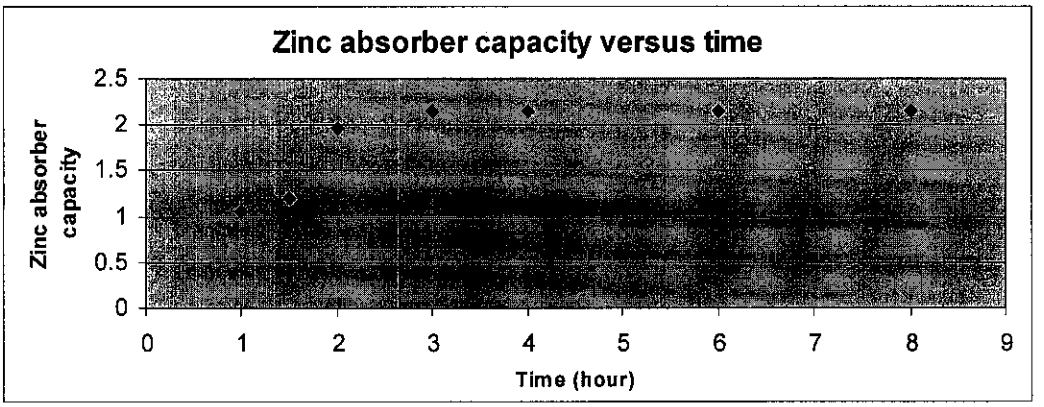


Figure 5.10: Zinc absorber capacity for chitosan carbon nanotube gel treated at 40 degrees Celsius.

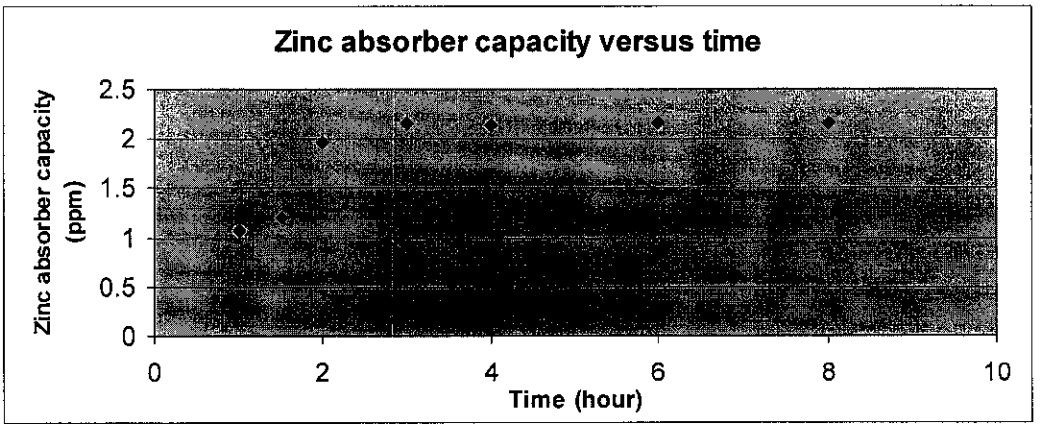


Figure 5.11: Zinc adsorption capacity for chitosan carbon nanotube gel treated at 50 degrees Celsius.

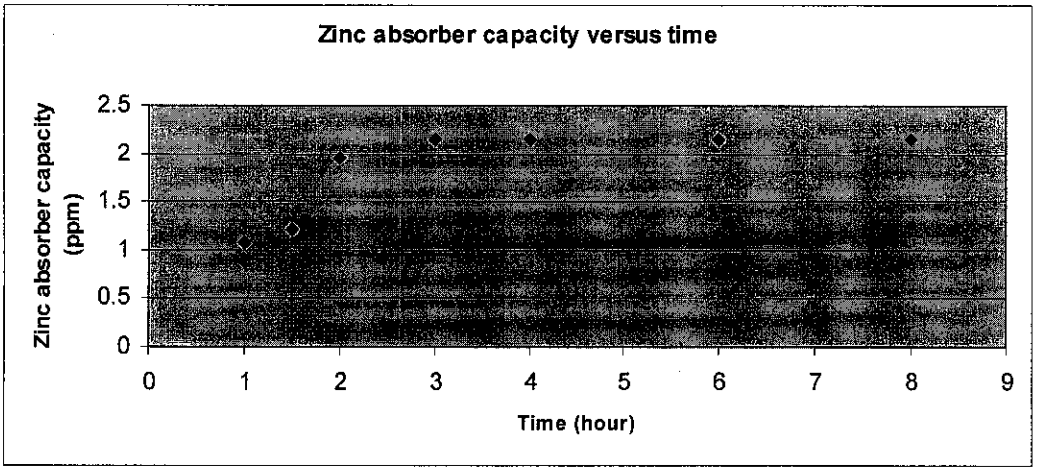


Figure 5.12: Zinc adsorption capacity for chitosan carbon nanotube gel treated at 60 degrees Celsius.

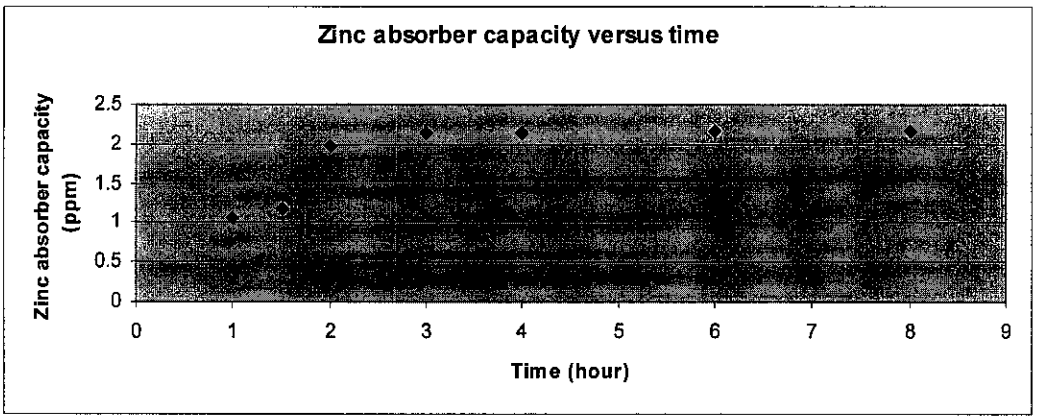


Figure 5.13: Zinc adsorption capacity for chitosan carbon nanotube gel treated at 70 degrees Celsius.

5.5 Percentage of ions uptake by adsorbent

By referring to figure 5.14, we can see clearly that total of ions uptake by the chitosan-CNT gel is more compared to the chitosan and chitosan gel. As being discussed before, the presence of carbon nanotube inside the gel had increased the porosity of the gel in adsorbing metal ions.

Zink ions uptake by the chitosan-CNT gel is less compared to chitosan gel and chitosan since the adsorbent are more likely to adsorbed Plumbum ions rather than zinc ions. This is might be because of other interference effect such as concentration of other metal ions which had a higher concentration than zinc ions.

In order to see the trend of total metal ions adsorbed by the chitosan, chitosan gel and chitosan-CNT gel, Figure 5.15, 5.16, and 5.17 was plotted which reflect to percentage of metal ions uptake by the adsorbent. Comparing to the graph, chitosan – CNT gel seems to be most efficient in adsorbing metal ions compared to chitosan and chitosan gel.

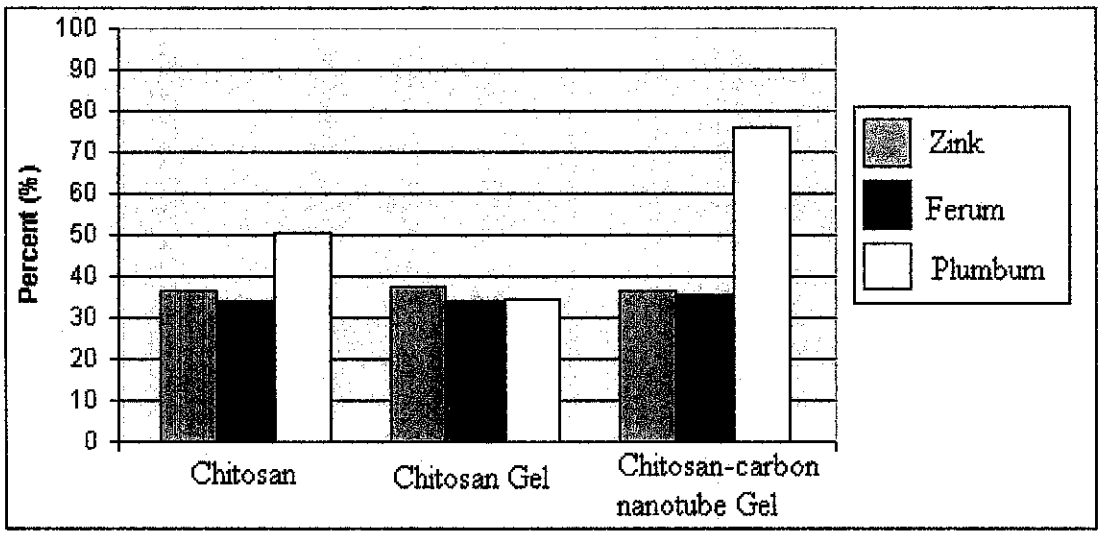


Figure 5.14: Percent of ions uptake by the adsorbent

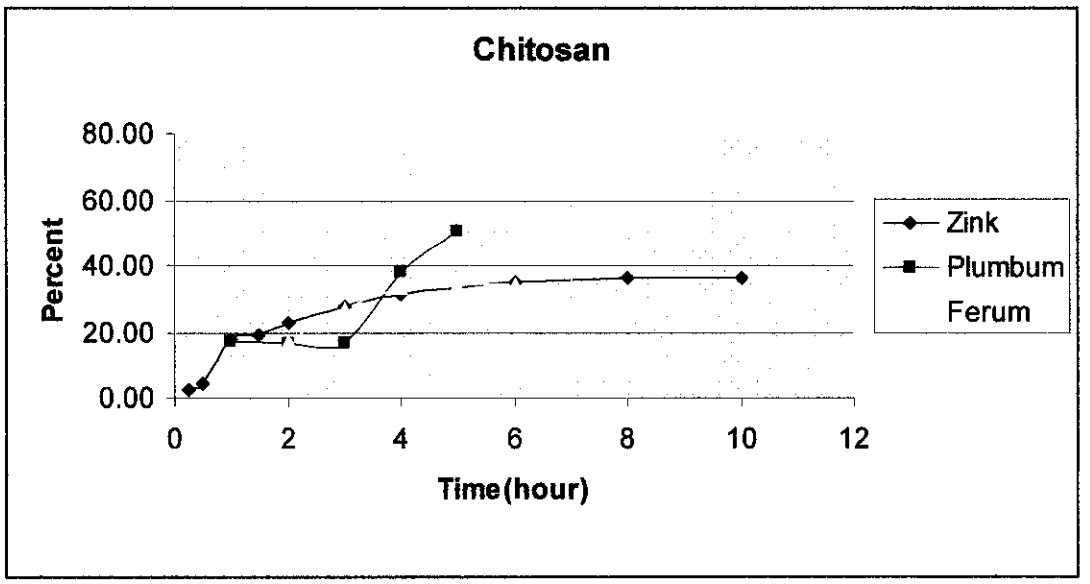


Figure 5.15: Chitosan adsorption percentage

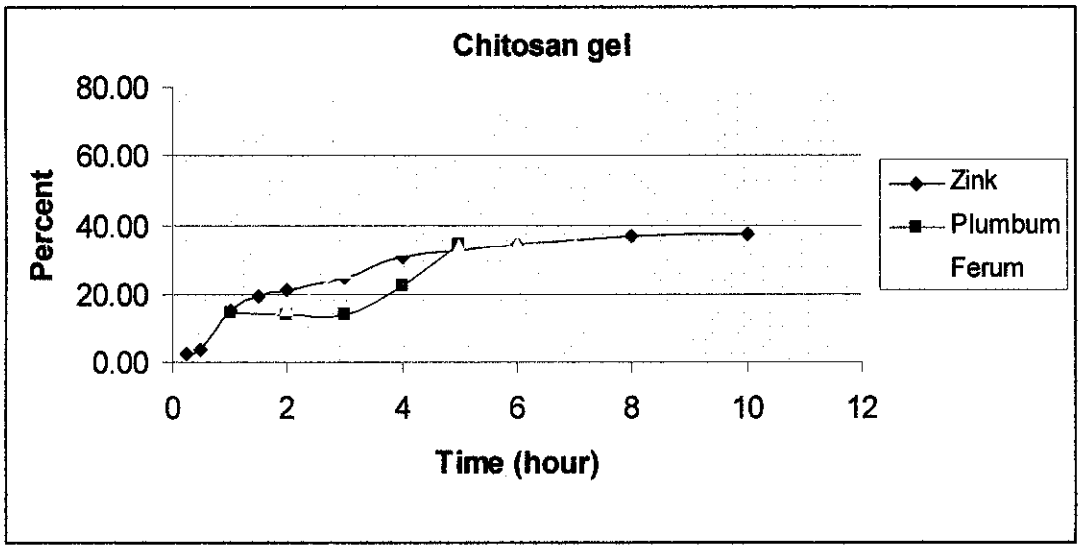


Figure 5.16: Chitosan gel adsorption percentage

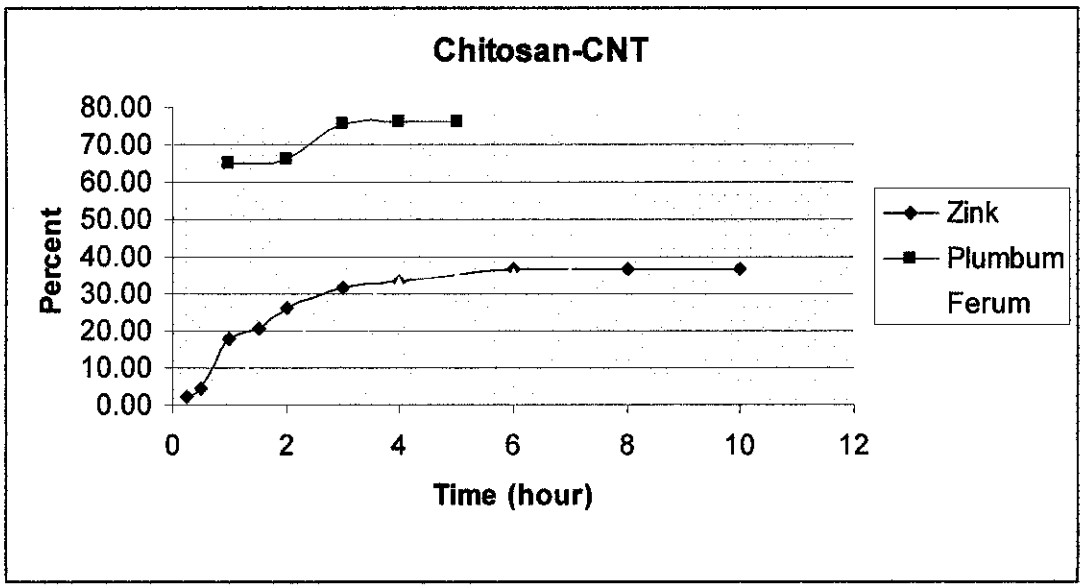


Figure 5.17: Chitosan-CNT adsorption percentage

CHAPTER 6
CONCLUSION

Removal of certain metal ions from waste water by using biopolymers is proved to be a great alternative at the moment. Chitosan is one of the best examples in removing heavy metal ions.

From the project which had been done, the all the objectives had been achieved, the first part of the objectives was to extract chitosan from crab shells and determine weight percentage of biopolymers that can be obtained. From the project, 70.81 gram of chitosan which is equivalent to 71 weight percent had been obtained from 197 gram of crab shell powder. Theoretically, the optimum amount of chitosan can be extracted from the chitin was about 70%. A part form that, the extraction method had precisely meet the target.

However, some modification should be made to the chitosan in order to improve its characteristic especially on the physical properties of the chitosan. Some test had been carried out in order to find the significant condition that had been applied to the adsorbent. The porosity of the absorber can be improved by adding some additive to the absorber, in this project, the additive used was carbon nanotube.

From the study, carbon nanotube – chitosan gel gave an improvement in total of metal ions adsorbed, which is about 16 percent better than chitosan and 91 percent better than chitosan gel .particularly, chitosan-CNT gel had adsorbed 76.1 percent of Pb^{2+}, 36.51 percent of Zn^{2+} and 35.46 percent of Fe^{2+} compared to chitosan and chitosan gel which only be able to adsorbed 50.57 percent of Pb^{2+}, 36.49 percent of Zn^{2+}, 34.08 percent of Fe^{2+} and 34.36 of Pb^{2+}, 37.5 percent of Zn^{2+}, 34.02 percent of Fe^{2+}. But hence, the efficiency can be better improved by using some cross linking agent in creating a more efficient absorber.

CHAPTER 7
RECOMMENDATIONS

From the experiment which had been done through out the project, there are some recommendation that can be made in order to improve the result obtained the project. It recommended that UTP will provided sufficient equipment for the FYP student in order to make sure student can do their project smoothly. Due to the time constrain and budget limitation, limited sample analyses are able to be tested. Another difficulty happen when a lot of students need to use the equipment, they had to wait for their turn before they can use that equipment.

Therefore, there are some recommendations that can be applied in order to increase the efficiency of the study and for the experiment:

- Further study on effect of cross link agent toward metal ions absorption rate. This can be conducted by treated the chitosan gel with several cross linking agent in order to improve the porosity of the absorber.
- Provide more privilege to the final year student in doing their project. Final year student should been given priority in using any equipment available. Some final year student need to share equipment with ETP student. Come to worst, they need to wait their turn before they can use the equipment.

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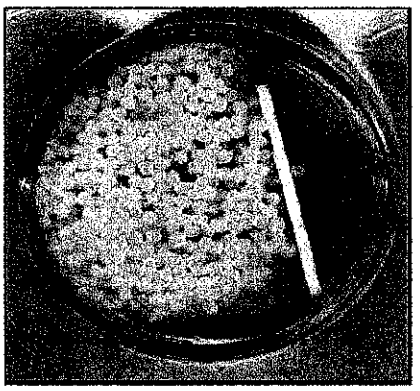
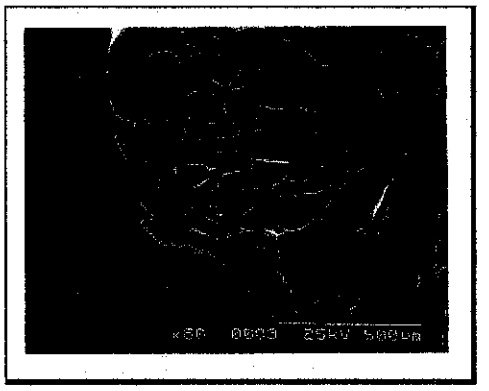
Appendix A

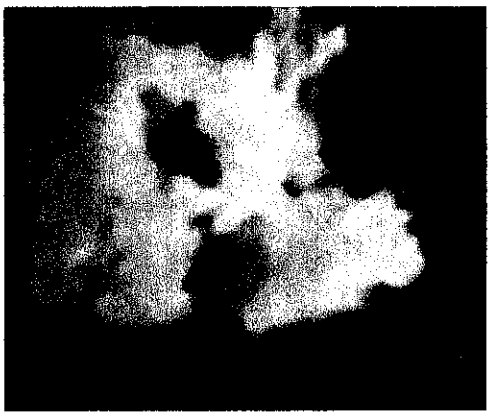


Overall view of chitosan beads.



Magnified cross section view of cross linked chitosan

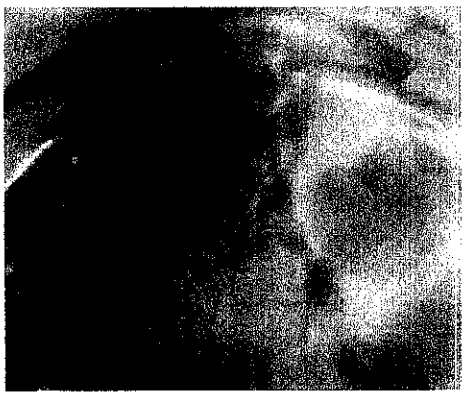
Chitosan – carbon nanotube gel

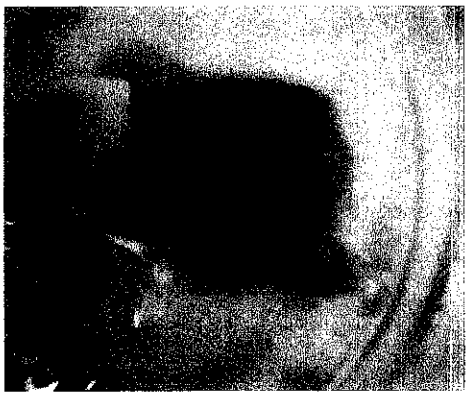


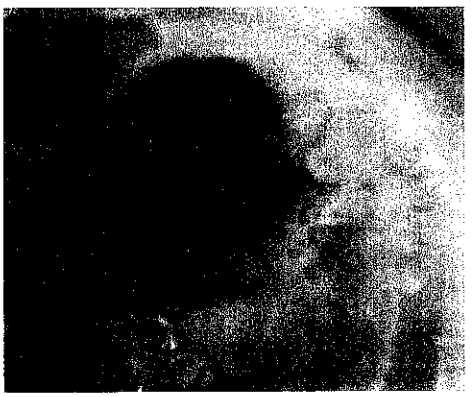
Chitosan gel

Chitosan – carbon nanotube gel
After being treated with waste water

Chitosan gel after being treated
with waste water

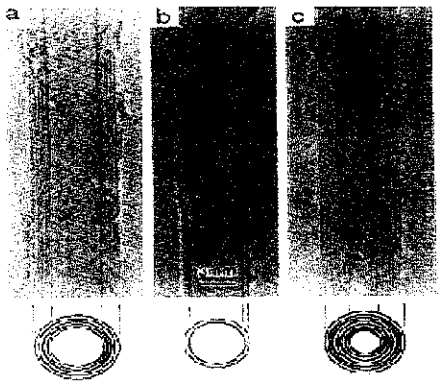




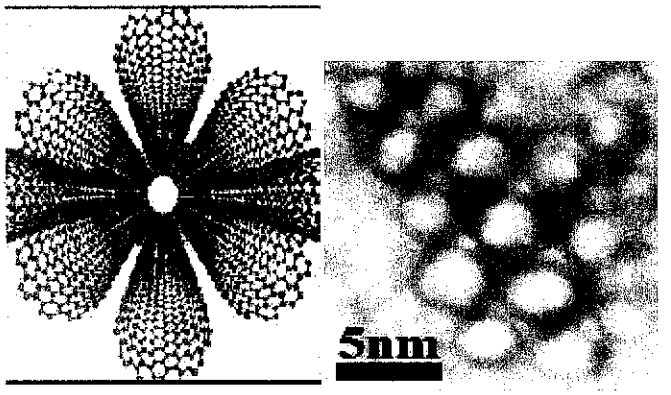


Chitosan carbon nanotube gel treated at 40,50,60,70 degrees Celsius.

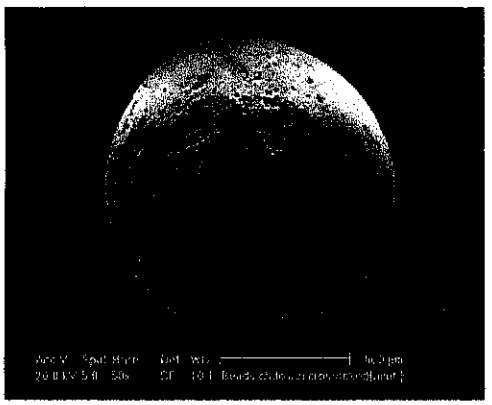
Appendix B



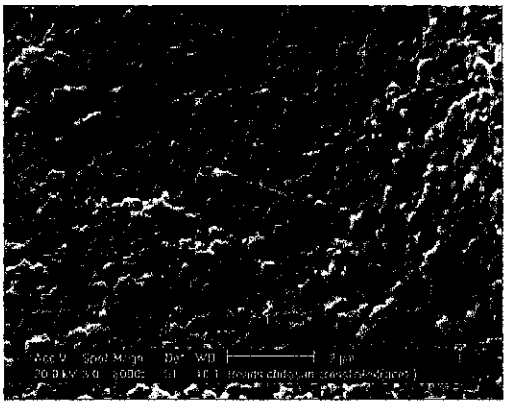
Multi wall nanotube, first observe by TEM, S. Iijima NEC Nature 1991



Bundle of single wall nanotubes



(a)



(b)

SEM photomicrographs: (a) whole chitosan microsphere at 50X; (b) external surface of microsphere at 8000X

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	6.3489	NONE
	5.9695	NONE
	5.3031	NONE
	4.7739	NONE
	4.3203	NONE
	4.1859	NONE
	4.1551	NONE
	4.1369	NONE
	5.9863	NONE
	5.4081	NONE
	4.7291	NONE
	4.3007	NONE
	4.2447	NONE
	4.2167	NONE
	4.2293	NONE
	6.0535	NONE
	5.3003	NONE
	4.6591	NONE
	4.2657	NONE
	4.2069	NONE
	4.1999	NONE
	4.2251	NONE

Sample ID	Pb:Flame Actual	Pb:Flame Actual
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	24.8348	NONE
	40.5887	NONE
	32.6811	NONE
	32.7089	NONE
	32.3530	NONE
	19.5075	NONE
	9.9706	NONE
	13.6463	NONE
	29.9619	NONE
	29.8117	NONE
	29.6282	NONE
	35.0834	NONE
	13.2904	NONE
	12.7399	NONE
	9.1865	NONE
	9.0253	NONE
	9.0419	NONE

3.3nm)

nt:

c

ımeters

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Width(nm):	1.0
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ner Angle(degree):	0

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o Intercept:	Yes
ıc. Unit:	ppm
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›gration Time (sec):	5
›ponse Time:	1

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ındard	2	3	5.00	0.00000
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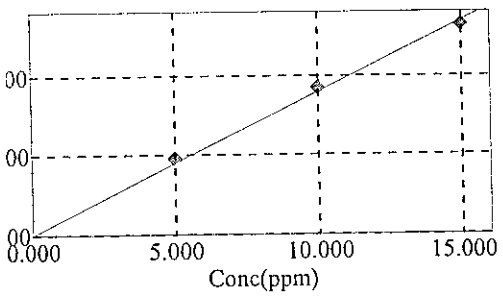

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15.0000	0.2646

98Conc+ 0

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TD Average

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Date	Time
5/11/2005	12:51 PM

STD Average

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5.0000	0.2646	0.0004	R4

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Date	Time
5/11/2005	12:52 PM

verage

Abs.	BG	Pos.
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%RSD	SD
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Date	Time
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VF	DF	CF	ActualConc.	
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Date	Time			
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Average

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Date	Time			
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Average

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	40.5887	0.7299	0.0034	3
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	0.5038	0.003677		
Date	Time			
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Average

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	32.6811	0.5877	0.0036	4
VF	DF	CF	ActualConc.	
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Unit	%RSD	SD		
	1.1069	0.006505		
Date	Time			
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Average

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	32.7089	0.5882	0.0044	5
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Unit	%RSD	SD
	0.6563	0.003818

Date	Time
5/11/2005	12:58 PM

Average

Conc.	Abs.	BG	Pos.
19.5075	0.3508	0.0055	7

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Unit	%RSD	SD
	1.3104	0.004596

Date	Time
5/11/2005	12:59 PM

Average

Conc.	Abs.	BG	Pos.
9.9706	0.1793	0.0053	8

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Unit	%RSD	SD
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Date	Time
5/11/2005	1:00 PM

Average

Conc.	Abs.	BG	Pos.
13.6463	0.2454	0.0056	9

VF	DF	CF	ActualConc.
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Unit	%RSD	SD
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Date	Time
5/11/2005	1:01 PM

Average

Conc.	Abs.	BG	Pos.
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Date	Time
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Average

	Abs.	BG	Pos.
	-0.0008	0.0063	R1

	%RSD	SD
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Date	Time
5/11/2005	1:02 PM

/erage

	Conc.	Abs.	BG	Pos.
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	VF	DF	CF	ActualConc.
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Date	Time
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verage

	Conc.	Abs.	BG	Pos.
	29.6282	0.5328	0.0068	12

	VF	DF	CF	ActualConc.
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Date	Time
5/11/2005	1:04 PM

Average

	Conc.	Abs.	BG	Pos.
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Date	Time
5/11/2005	1:05 PM

Average

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. Unit	%RSD	SD	
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Date	Time
5/11/2005	1:06 PM

. Average

	Conc.	Abs.	BG	Pos.
	12.7399	0.2291	0.0072	15

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c. Unit	%RSD	SD	
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Date	Time
5/11/2005	1:06 PM

/erage

		Abs.	BG	Pos.
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Date	Time
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.verage

	Conc.	Abs.	BG	Pos.
	9.1865	0.1652	0.0073	16

	VF	DF	CF	ActualConc.
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Unit	%RSD	SD	
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Date	Time
5/11/2005	1:08 PM

Average

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Unit	%RSD	SD	
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Date	Time
5/11/2005	1:09 PM

Average

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. Unit	%RSD	SD	
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Date	Time
5/11/2005	1:10 PM

3.3nm)

nt:

e

ımeters

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ɔ Intercept:	Yes
ıc. Unit:	ppm
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gration Time (sec):	5
ponse Time:	1

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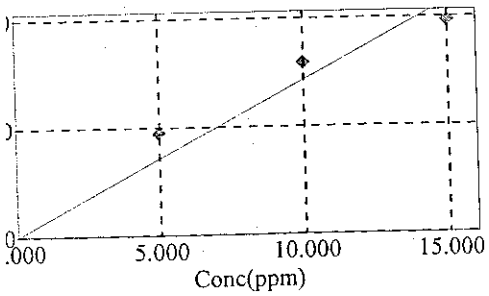
slope	2	3	5.00	0.00000

Parameters

Type	Judge Calc.	Standard Value	Out of Control Remark

ırve (C# : 01)



Conc (ppm)	Abs
5.0000	0.4708
10.0000	0.7930
15.0000	0.9811

iConc+ 0

D Average

/alue	Abs.	BG	Pos.
00	0.4708	0.0024	R2

%RSD	SD
0.0751	0.000354

Date	Time
5/12/2005	2:43 PM

'D Average

Value	Abs.	BG	Pos.
)000	0.7930	0.0035	R3

%RSD	SD
0.5261	0.004172

Date	Time
5/12/2005	2:44 PM

TD Average

: Value	Abs.	BG	Pos.
.0000	0.9811	0.0047	R4

%RSD	SD
0.0721	0.000707

Date	Time
5/12/2005	2:44 PM

verage

	Abs.	BG	Pos.
	-0.0007	-0.0014	R1

%RSD	SD
20.2030	0.000141

Date	Time
5/12/2005	2:45 PM

.verage

	Conc.	Abs.	BG	Pos.
	6.4707	0.4622	0.0008	1

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	6.4707

Unit	%RSD	SD	
	0.5660	0.002616	

Date	Time
5/12/2005	2:46 PM

Average

	Conc.	Abs.	BG	Pos.
	6.3489	0.4535	0.0004	2

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	6.3489

Unit	%RSD	SD	
	0.5301	0.002404	

Date	Time
5/12/2005	2:47 PM

Average

	Conc.	Abs.	BG	Pos.
	5.9695	0.4264	-0.0004	3

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	5.9695

. Unit	%RSD	SD	
	0.1824	0.000778	

Date	Time
5/12/2005	2:48 PM

Average

	Conc.	Abs.	BG	Pos.
	5.3031	0.3788	-0.0014	4

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	5.3031

:. Unit	%RSD	SD	
	0.2054	0.000778	

Date	Time
5/12/2005	2:49 PM

. Average

	Conc.	Abs.	BG	Pos.
	4.7739	0.3410	-0.0018	5

	VF	DF	CF	ActualConc.
)	1.00	1.00	1.000000	4.7739

c. Unit	%RSD	SD	
	1.2651	0.004313	

Date	Time
5/12/2005	2:49 PM

(Average

	Conc.	Abs.	BG	Pos.
	4.3203	0.3086	-0.0007	6

	VF	DF	CF	ActualConc.
0	1.00	1.00	1.000000	4.3203

ıc. Unit	%RSD	SD	
	1.0309	0.003182	

Date	Time
5/12/2005	2:50 PM

Average

	Conc.	Abs.	BG	Pos.
	4.1859	0.2990	-0.0004	7

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.1859

Unit	%RSD	SD	
	0.6622	0.001980	

Date	Time
5/12/2005	2:51 PM

Average

	Conc.	Abs.	BG	Pos.
	4.1551	0.2968	-0.0008	8

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.1551

Unit	%RSD	SD	
	1.2629	0.003748	

Date	Time
5/12/2005	2:52 PM

Average

	Conc.	Abs.	BG	Pos.
	4.1369	0.2955	-0.0007	9

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.1369

Unit	%RSD	SD	
	0.8615	0.002546	

Date	Time
5/12/2005	2:53 PM

Average

	Conc.	Abs.	BG	Pos.
	5.9863	0.4276	0.0003	10

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	5.9863

Unit	%RSD	SD	
	0.9922	0.004243	

Date	Time
5/12/2005	2:53 PM

Average

		Abs.	BG	Pos.
		-0.0071	-0.0007	R1

	%RSD	SD	
	11.9511	0.000849	

Date	Time
5/12/2005	2:54 PM

Average

	Conc.	Abs.	BG	Pos.
	5.4081	0.3863	0.0010	11

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	5.4081

Unit	%RSD	SD	
	1.0251	0.003960	

Date	Time
5/12/2005	2:55 PM

Average

	Conc.	Abs.	BG	Pos.
	4.7291	0.3378	0.0003	12

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.7291

Unit	%RSD	SD	
	0.5651	0.001909	

Date	Time
5/12/2005	2:56 PM

Average

	Conc.	Abs.	BG	Pos.
	4.3007	0.3072	-0.0004	13

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.3007

Unit	%RSD	SD	
	0.0230	0.000071	

Date	Time
5/12/2005	2:57 PM

Average

	Conc.	Abs.	BG	Pos.
	4.2447	0.3032	-0.0001	14

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.2447

Unit	%RSD	SD	
	1.0493	0.003182	

Date	Time
5/12/2005	2:57 PM

Average

	Conc.	Abs.	BG	Pos.
	4.2167	0.3012	-0.0003	15

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.2167

Unit	%RSD	SD	
	1.2677	0.003818	

Date	Time
5/12/2005	2:58 PM

Average

	Conc.	Abs.	BG	Pos.
	4.2293	0.3021	0.0000	16

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	4.2293

Unit	%RSD	SD	
	0.7022	0.002121	

Date	Time
5/12/2005	2:59 PM

Average

	Conc.	Abs.	BG	Pos.
	6.0535	0.4324	0.0008	17

	VF	DF	CF	ActualConc.
	1.00	1.00	1.000000	6.0535

Unit	%RSD	SD	
	0.2126	0.000919	

Date	Time
5/12/2005	3:00 PM

)NK Average

	Conc.	Abs.	BG	Pos.
	5.3003	0.3786	0.0004	18

	VF	DF	CF	ActualConc.
)000	1.00	1.00	1.000000	5.3003

	%RSD	SD	
Conc. Unit	0.5230	0.001980	
:			

	Date	Time
	5/12/2005	3:01 PM

UNK Average

	Conc.	Abs.	BG	Pos.
	4.6591	0.3328	0.0008	19

	VF	DF	CF	ActualConc.
)0000	1.00	1.00	1.000000	4.6591

	%RSD	SD	
lConc. Unit	0.7011	0.002333	
E			

	Date	Time
	5/12/2005	3:01 PM

UNK Average

	Conc.	Abs.	BG	Pos.
	4.2657	0.3047	0.0010	20

	VF	DF	CF	ActualConc.
)00000	1.00	1.00	1.000000	4.2657

	%RSD	SD	
alConc. Unit	1.2067	0.003677	
IE			

	Date	Time
	5/12/2005	3:02 PM

.K Average

		Abs.	BG	Pos.
		-0.0092	-0.0005	RI

	%RSD	SD	
	0.7728	0.000071	

	Date	Time
	5/12/2005	3:03 PM

. : UNK Average

	Conc.	Abs.	BG	Pos.
	4.2069	0.3005	0.0004	21

	VF	DF	CF	ActualConc.
:				
.000000	1.00	1.00	1.000000	4.2069

	%RSD	SD	
tualConc. Unit	0.8471	0.002546	
)NE			

	Date	Time
	5/12/2005	3:04 PM

2 : UNK Average

	Conc.	Abs.	BG	Pos.
-	4.1999	0.3000	0.0014	22

	VF	DF	CF	ActualConc.
F				
1.000000	1.00	1.00	1.000000	4.1999

	%RSD	SD	
ctualConc. Unit	0.1886	0.000566	
ONE			

	Date	Time
#		
I	5/12/2005	3:05 PM

NK Average

	Conc.	Abs.	BG	Pos.
	4.2251	0.3018	0.0015	23
	VF	DF	CF	ActualConc.
000	1.00	1.00	1.000000	4.2251
Conc. Unit	%RSD	SD		
	0.6325	0.001909		
Date	Time			
5/12/2005	3:06 PM			